

Exhibit 4



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(12) **United States Patent**
Davis, Jr.

(10) Patent No.: **US 7,208,605 B2**
(45) Date of Patent: **Apr. 24, 2007**

(54) **FUNCTIONALIZED IONIC LIQUIDS, AND METHODS OF USE THEREOF**

FOREIGN PATENT DOCUMENTS

JP 54079278 A * 6/1979

OTHER PUBLICATIONS

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(73) Assignee: University of South Alabama, Mobile, AL (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 418 days.

Fargo-Duke et al., Catalyzed Isomerizations in Room Temperature Ionic Liquids with Acidic Counteranion as Recyclable Reaction Media, *Catalysis Communications*, 3 2002, 185-190.*
Visser et al., Hydrophobic Ionic Liquids Incorporating N-alkylpyrrolidinium Cations and their Utilization in Liquid-Liquid Separations, *Chem Commun.* 2001, 2484-2485.*
Welton, Room-Temperature Ionic Liquids: Solvents for Synthesis and Catalysis, *Chem. Rev.* 1997, 99, 2071-2083.*

(Continued)

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(58) Field of Classification Search 548/110; 564/291, 295; 568/10, 11, 9

See application file for complete search history.

(56) References Cited

U.S. PATENT DOCUMENTS

4,761,164 A 8/1988 Fox et al. 56/16
2002/0010291 A1* 1/2002 Murphy 526/133

(57) **ABSTRACT**

One aspect of the present invention relates to ionic liquids comprising a pendant Brønsted-acidic group, e.g., a sulfonic acid group. Another aspect of the present invention relates to the use of an ionic liquid comprising a pendant Brønsted-acidic group to catalyze a Brønsted-acid-catalyzed chemical reaction. A third aspect of the present invention relates to ionic liquids comprising a pendant nucleophilic group, e.g., an amine. Still another aspect of the present invention relates to the use of an ionic liquid comprising a pendant nucleophilic group to catalyze a nucleophile-assisted chemical reaction. A fifth aspect of the present invention relates to the use of an ionic liquid comprising a pendant nucleophilic group to remove a gaseous impurity, e.g., carbon dioxide, from a gas, e.g., sour natural gas.

24 Claims, 1 Drawing Sheet

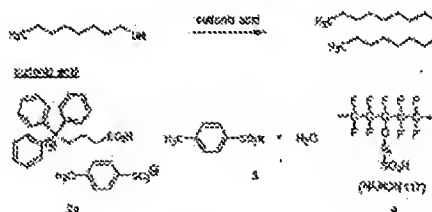


TABLE 1. Analysis of Graft Ester Formation.

Entry	Sulfonic Acid Derivative	Ratio of 1-octanol to sulfonic acid derivative	Ratio of 1-octanol to water	Yield, %
1	2a	1.2:1.0	54.65	25
2	2a	1.5:1.0	20.23	59
3	2a	3.0:1.0	37.25	18
4	3	1.2:1.0	15.85	49
5	4	3	26.4	3

*Ratio based upon unreacted 1-octanol to individual graft ester (GC analysis of the distilled product). **Yield may which is based upon amount of 1-octanol (0.5 mL, 3.17 mmol) and HAPION (314 mg, 0.28 mmol (0.55 mmol/g) used.

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FUNCTIONALIZED IONIC LIQUIDS, AND
METHODS OF USE THEREOF

RELATED APPLICATIONS

This application claims the benefit of priority to U.S. Provisional Patent Application Ser. No. 60/370,130, filed Apr. 5, 2002.

BACKGROUND OF THE INVENTION

Ionic Liquids

Room temperature ionic liquids consist of ions. However, unlike conventional molten salts (for example, molten sodium chloride), these materials often melt below 100° C. Since the melting points are low, ionic liquids can act as solvents in which reactions can be performed, and because the liquid is made of ions rather than molecules, such reactions often provide distinct selectivities and reactivities as compared to conventional organic solvents.

Room-temperature ionic liquids have been used as clean solvents and catalysts for green chemistry and as electrolytes for batteries, photochemistry and electrosynthesis. They have no significant vapor pressure and thus create no volatile organic contaminants. They also allow for easy separation of organic molecules by direct distillation without loss of the ionic liquid. Their liquid range can be as large as 300° C. allowing for large reaction kinetic control, which, coupled with their good solvent properties, allows small reactor volumes to be used. Salts based upon poor nucleophilic anions, such as $[\text{BF}_4]^-$, $[\text{PF}_6]^-$, $[\text{CF}_3\text{CO}_2]^-$, and $[\text{CF}_3\text{SO}_3]^-$, are water and air insensitive and possess remarkably high thermal stability. Many of these materials are based around an imidazolium cation, 1-alkyl-3-methylimidazolium. By changing the anion or the alkyl chain on the cation, a wide variation in properties, such as hydrophobicity, viscosity, density and solvation, can be obtained. For example, ionic liquids will dissolve a wide range of organic molecules to an appreciable extent, the solubility being influenced by the nature of the counter anion.

The unique physical properties of ionic liquids have been found to offer certain advantages in numerous applications. For example, U.S. Pat. No. 5,827,602 to Koch et al. discloses ionic liquids having improved properties for applications in batteries, electrochemical capacitors, catalysis, chemical separations, and other uses. The ionic liquids described in Koch et al. are hydrophobic in nature, being poorly soluble in water, and contain only non-Lewis acid anions. When fluorinated, they were found to be particularly useful as hydraulic fluids and inert liquid diluents for highly reactive chemicals. In addition, ionic liquids have been discussed by Freemantle, M. Chem. Eng. News 1998, 76 [March 30], 32; Carmichael, H. Chem. Britain, 2000, [January], 36; Seddon, K. R. J. Chem. Tech. Biotechnol. 1997, 68, 351; Welton, T. Chem. Rev. 1999, 99, 2071; Bruce, D. W., Rowles, C. I., Seddon, K. R. Chem. Commun. 1996, 1625; Merrigan, T. L., Bates, E. D., Dorman, S. C., Davis, J. H. Chem. Commun. 2000, 2051; Freemantle, M. Chem. Eng. News, 2000, 78 [May 15], 37. See also Holbrey, J. D.; Seddon, K. R. Clean Products and Processes, 1999, 1, 223-236; and Dupont, J., Consani, C. S. Spenser, J. J. Braz. Chem. Soc. 2000, 11, 337-344.

Ionic liquids have been disclosed for use as solvents for a broad spectrum of chemical processes. These ionic liquids, which in some cases can serve as both catalyst and solvent, are attracting increasing interest from industry because they

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promise significant environmental benefits, e.g., because they are nonvolatile they do not emit vapors. Hence, for example, they have been used in butane dimerization processes, WO 95/21871, WO 95/21872 and WO 95/21806 relate to ionic liquids and their use to catalyze hydrocarbon conversion reactions such as polymerization and alkylation reactions. The ionic liquids described for this process were preferably 1-($\text{C}_1\text{--}\text{C}_4$ alkyl)-3-($\text{C}_6\text{--}\text{C}_{10}$ alkyl) imidazolium chlorides and especially 1-methyl-3- C_{10} alkyl-imidazolium chloride, or 1-hydrocarbyl pyridinium halides, where the hydrocarbyl group is, for example, ethyl, butyl or other alkyl. PCT publication WO 01/25326 to Lamanna et al. discloses an antistatic composition comprising at least one ionic salt consisting of a nonpolymeric nitrogen cation and a weakly coordinating fluorine anion, the conjugate acid of the anion being a superacid, in combination with thermoplastic polymer. The composition was found to exhibit good antistatic performances over a wide range of humidity levels.

Bronsted Acid Catalysis

From undergraduate laboratories to chemical manufacturing plants, the use of strong Bronsted acids is ubiquitous. Smith, M. B.; March, J. *March's Advanced Organic Chemistry*; Wiley-Interscience: New York, 2001; Chapter 8. In this context, solid acids are being more widely used since, as non-volatile materials, they are deemed less noxious than traditional liquid acids. Ritter, S. K. Chem. Eng. News, 2001, 79 (40), 63-67. However, solid acids have shortcomings. Among the more troublesome of these are restricted accessibility of the matrix-bound acidic sites, high m.w./active site ratios, and rapid deactivation from coking. Ishihara, K.; Hasegawa, A. and Yamamoto, H. *Angew. Chem. Int. Ed.*, 2001, 40, 4077-4079; and Hammer, M. A. and Sun, Q. *Appl. Catal. A: General*, 2001, 221, 45-62.

Bearing in mind both the advantages and disadvantages of solid acids, the search continues for systems that are Bronsted acids with solid-like non-volatility but that manifest the mobility, greater effective surface area and potential activity of a liquid phase. Combining just these characteristics, ionic liquids (IL) have been described as one of the most promising new reaction mediums. Seddon, K. R. J. Chem. Technol. Biotechnol. 1997, 68, 351-356. Not only can these unusual materials dissolve many organic and inorganic substrates, they are also readily recycled and are tunable to specific chemical tasks. Bates, E. D.; Mayton, R. D.; Nair, I. and Davis, J. H. Jr. *J. Am. Chem. Soc.* 2002, 124, 926-927; Visser, A. E.; Holbrey, J. D.; Rogers, R. D. *Chem. Commun.*, 2001, 2484-2485; Visser, A. E.; Swatoski, R. P.; Reichert, W. M.; Mayton, R.; Shell, S.; Wierzbicki, A.; Davis, J. H. Jr.; Rogers, R. D. *Chem. Commun.*, 2001, 135-136; Merrigan, T. L.; Bates, E. D.; Dorman, S. C.; Davis, J. H. Jr. *Chem. Commun.*, 2000, 2051-2052; Forrester, K. J.; Davis, J. H. Jr. *Tetrahedron Lett.*, 1999, 40, 1621-1622; and Morrison, D. W.; Forbes D. C.; Davis, J. H. Jr. *Tetrahedron Letters*, 2001, 42, 6053-6057.

Further, the chemical industry is under significant pressure to replace the volatile organic compounds that are currently used as solvents in organic synthesis. Many of these solvents, such as chlorinated hydrocarbons, are toxic and hazardous for the environment, due to their emissions in the atmosphere and the contamination of aqueous effluents. Ionic liquids (IL) seem to offer a solution to this problem, too. Ionic liquids have no measurable vapor pressure. This means that they don't evaporate, and therefore they emit no hazardous vapors in the atmosphere, and replenishing of the solvent is not required. This property also allows easy

Exhibit 5

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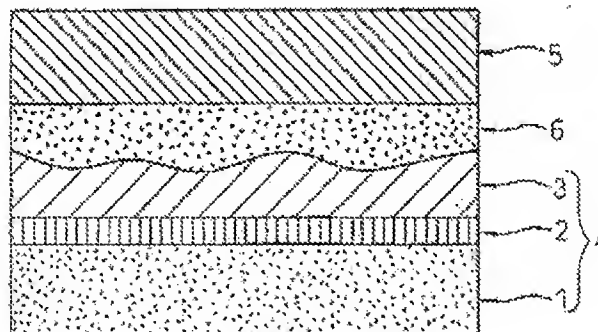
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[Continued on next page]

(84) Title: ELECTROLYTE FOR PHOTOVOLTAIC DEVICE AS WELL AS PHOTOVOLTAIC DEVICE AND DYE-SENSE-
TIZED SOLAR CELL INCLUDING THAT ELECTROLYTE



(57) Abstract: An electrolyte for a photovoltaic device including (i) a layered clay mineral and/or an organically modified layered clay mineral and (ii) an ionic liquid as well as a photovoltaic device including a photoelectrode including a transparent conducting layer and a metal oxide semiconductor mesoporous film using, as an electrolyte layer, the same, a counter electrode facing this photoelectrode and an electrolyte layer arranged between the photoelectrode and the counter electrode as well as a dye-sensitized solar cell composed of a photovoltaic device and a photosensitizer coated on a metal oxide semiconductor mesoporous film of the photovoltaic device, wherein the conductive substrate is obtained by coating, on a conductive substrate, a conductive polyaniline dispersion stably dispersed in an organic solvent including (A) a polyaniline obtained by polymerization of aniline or an aniline derivative, (B) a sulfonic acid compound and/or (C) an organic polymer having a protonic acid group, (D) a molecular weight modifier, and (E) an organic solvent capable of dissolving the sulfonic acid compound (B), the organic polymer having a protonic acid group (C), and the molecular weight modifier (D).

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onium ions, more preferably 0.5 to 1.5 weight times, based upon the preferable amount of anion exchange of the layered clay mineral, preferably at a temperature of 10 to 95°C.

5 The ionic liquid usable in the first and second aspects of the present invention is not particularly limited. It is possible to use any ionic liquid used as an electrolyte in the past. Specifically, those described in Ono Hiroyuki ed., *Ionic Liquid - Front Edge and Future*
 10 *of Development* -, edited by Hiroyuki Ono, CMC Shuppan (2003), *Creation of Functions and Applications of Ionic Liquids*, NTS (2004), etc., quaternary ammonium salts, imidazolium salts, pyridinium salts, pyrrolidinium salts, piperidinium salts, etc. (As the salt, for example, a
 15 salt comprised of I^- , BF_4^- , PF_6^- , CF_3COO^- , $CF_3SO_3^-$, $N(CF_3SO_2)_2^-$, $N(CN)_2^-$, $C(CN)_3^-$, and other anions may be exemplified) may be mentioned. As preferable ionic liquids, there are aliphatic quaternary ammonium salts, imidazolium salts, pyridinium salts, and pyrrolidinium
 20 salts. Particularly preferable are imidazolium salts shown in the following formula (III).



(III)

wherein R^5 indicates a methyl group or ethyl group, R^6 indicates a C_1 to C_8 hydrocarbon group and X indicates an
 25 anion such as I^- , BF_4^- , PF_6^- , CF_3COO^- , $CF_3SO_3^-$, $N(CF_3SO_2)_2^-$, $N(CN)_2^-$, $C(CN)_3^-$, etc.

When an electrolyte produced from (a) a layered clay mineral and/or an organically modified layered clay mineral or (b) a swelled dispersion of a layered clay
 30 mineral and/or an organically modified layered clay mineral and an ionic liquid according to the present invention is used as a photovoltaic device electrolyte, a

Exhibit 6

イオン性液体

—開発の最前線と未来—

Ionic Liquids: The Front and Future of Material Development

監修：大野弘幸 *Supervisor: Hiroyuki Ohno*

シーエムシー出版

図1 イオン性液体の高分子化

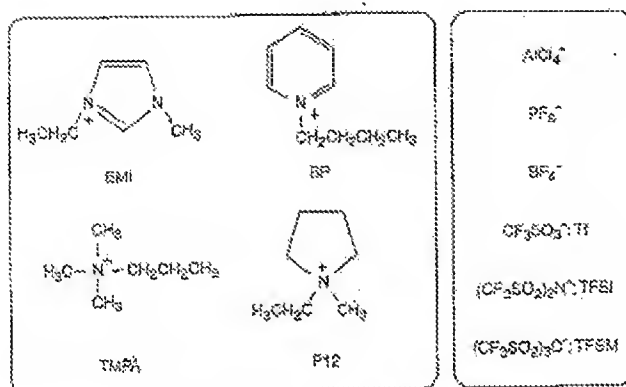


図1 代表的なイオン性液体の構造とその符号

に提出された代表的なイオン性液体の構造を示す。

イオン性液体の一番の特徴は、イオン性物質でありながら融点が著しく低いことである。物質の融点 (T_m) は ΔH_m を溶解エンタルピー変化、 ΔS_m を溶解エントロピー変化としたとき

$$T_m = \Delta H_m / \Delta S_m$$

で与えられるため、 ΔH_m の低下、 ΔS_m の増大が融点低下をもたらす。イオン性液体を考えた場合ではカチオンとアニオンのサイズが比較的大きいこと、カチオンやアニオン中の電荷が非局在化していることが多い。これはイオン性化合物の ΔH_m の小さな相互作用であるクーロン力を弱減させるためと考えられる。またカチオンとアニオンのコンフォメーションの自由度が高いこと、さらにカチオンやアニオンの構造が非対称であることなどは、 ΔH_m の増大を抑制し、融点の低下をもたらすと考えられている。一例として図2に、BMIカチオンの対アニオン構造の違いが室温での状態をどのように変化させるかを耳貫で示す。対アニオンが PF_6^- の場合は室温で白色の結晶固体 ($T_m = 78^\circ\text{C}$) である。一方、 BF_4^- や TFSI^- アニオンの場合には無色透明な液体となり、融点はそれぞれ 16°C 、 -38°C にまで低下する。

イオン性液体の一般的な特徴として、上述した融点が低いこと以外に以下の点を挙げることもできる。

- ① イオンのみからなる新しい溶媒
- ② 液体でありながら腐食性がない (不揮発性)
- ③ 耐熱性が高く液体密度が低い (例えば図1に示したBMI/TFSIの耐熱性は 300°C 以上)

参考文献3

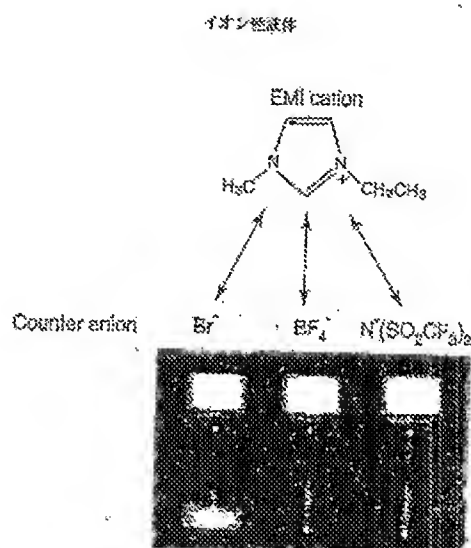


図2 対アニオンの構造の違いによるEMI塩の室温における外観の違い。

- ② 分解温度以下では不溶性
- ③ 化学的に安定
- ④ イオン導電性が高い（一例として図3にEMIBF₄、EPBPF₄のイオン導電率の温度依存性を示す）
- ⑤ 分解電圧が水などの溶媒と比べ高く電気分解しにくい（一例として図4にEMIBF₄、EPBPF₄のサイクリックボルタモグラムの示す）

このような特徴を生かし、

- ① 新しい有機合成、高分子合成の溶媒、とくに触媒反応の溶媒としての検討
 - ② 物質の分離、回収を目的とした安定で揮発性がなくリサイクル可能なグリーン溶媒としての検討
 - ③ 電気化学系に用いる新しい電解質（イオン伝導体）としての検討
- などが世界的に急速に進んでいる¹⁾²⁾。

1.3 イオン性液体中でのビニルモノマーのその場重合によるイオンゲルの合成

イオン伝導性の物質は、化学エネルギーと電気エネルギーの相互交換、あるいは化学情報と電気信号の相互変換を可能にする電気化学系を構築するために不可欠である。前述したイオン性液

イオン性液体

—開発の経緯と未来—

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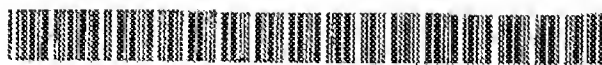
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Exhibit 7



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(54) ELECTROLYTE FOR RECHARGEABLE
LITHIUM BATTERY AND RECHARGEABLE
LITHIUM BATTERY COMPRISING SAME

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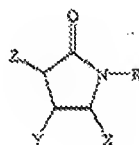
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(57) ABSTRACT

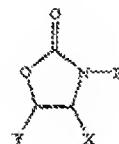
Disclosed is an electrolyte for a rechargeable lithium battery including a non-aqueous organic solvent, a lithium salt, and a lactam-based compound selected from the group consisting of compounds represented by formulas 1 to 6:



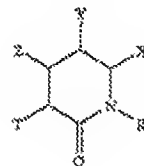
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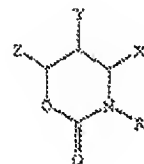
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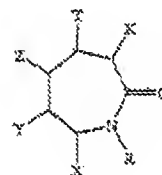
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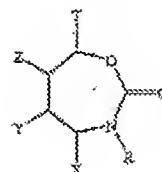
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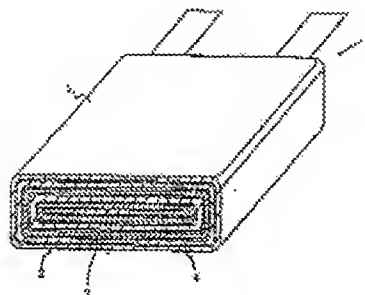
(5)



(6)



where R is a H radical, a C₁ to C₆ alkanyl or a halogen radical; and X, T, Y and Z are identically or independently H radicals or halogen radicals.

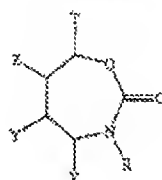


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Oct. 28, 2004

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(6)

[0019] where R is a hydrogen radical, a C_2 to C_6 alkanyl, preferably vinyl, or a halogen radical; and X, T, Y, and Z are identically or independently hydrogen radicals or halogen radicals.

[0020] The examples of the lactam-based compound may be N-vinylcaprolactam, N-bromocaprolactam, delta-valerolactam, N-methyl-gamma-caprolactam, vinylbutyrolactam, caprolactam, or gamma-butyrolactam.

[0021] The lactam-based compound is preferably provided in an amount between 0.01 and 10 wt % based on the weight of the non-aqueous organic solvent and more preferably, between 1 and 8 wt %. If the amount of the lactam-based compound is less than 0.01 wt %, the effect of the addition of the lactam-based compound cannot be obtained. If that of the lactam-based compound is more than 10 wt %, the cycle life characteristic deteriorates.

[0022] The non-aqueous organic solvent includes at least one cyclic carbonate, linear carbonate, ester, or ketone. If a mixture thereof is used, the mixing ratio can be suitably controlled according to the desired battery performance, as is well understood in the related art. Suitable cyclic carbonates include ethylene carbonate, propylene carbonate, and mixtures thereof. Linear carbonates include dimethyl carbonate, diethyl carbonate, ethylmethyl carbonate, and methyl propyl carbonate. Esters include gamma-butyrolactone, valerolactone, decanolide, and mevalolactone. A suitable ketone is polymethylvinyl ketone.

[0023] The non-aqueous organic solvent may further be a halogenated benzene in order to improve low-temperature, cycle life, and high-temperature characteristics. A halogenated benzene is represented by formula 7:



(7)

[0024] where X is a radical of F, Cl, Br or I, and n is an integer from 1 to 3.

[0025] The preferred amount of the halogenated benzene is 5 to 40 parts by volume based on 100 parts by volume of the total electrolyte. If the amount of the halogenated benzene is less than 5 parts by volume, ionic conductivity at low temperatures decreases. If that of the halogenated benzene is more than 40 parts by volume, ionic conductivity at room temperatures decreases.

[0026] The electrolyte includes a lithium salt as a supporting electrolytic salt. Suitable lithium salts include lithium hexafluorophosphate ($LiPF_6$), lithium tetrafluoroborate ($LiBF_4$), lithium perchlorate ($LiClO_4$), lithium trifluoromethanesulfonate (CF_3SO_3Li), and lithium hexafluoroarsenate ($LiAsF_6$). The lithium salt acts as a source for supplying lithium ions in the battery, and helps the working of the battery. In addition, the lithium salt activates transfer of lithium ions between a positive electrode and a negative electrode.

[0027] A rechargeable lithium battery with the inventive electrolyte includes a positive electrode and a negative electrode.

[0028] An embodiment of the rechargeable lithium battery 1 of the present invention is shown in FIG. 1. The rechargeable lithium battery 1 in FIG. 1 includes a positive electrode 3, a negative electrode 4, and an electrolyte 2. The positive electrode 3, the negative electrode 4, and the electrolyte 2 are received in a battery case 5.

[0029] The positive electrode includes a positive active material in which lithium intercalation reversibly occurs. Examples of the positive active material are lithium transition metal oxides such as $LiCoO_2$, $LiNiO_2$, $LiMnO_2$, LiM_2O_4 , or $LiNi_{1-x}Co_xM_yO_2$ where $0.5 \leq x \leq 1$, $0 \leq y \leq 1$, and M is a metal such as Al, Sr, Mg, or La.

[0030] The negative electrode includes a negative active material in which lithium intercalation reversibly occurs. Examples of negative active materials include crystalline or amorphous carbonaceous materials, or carbon composites.

[0031] The positive active material and the negative active material are respectively coated on current collectors to produce electrodes, and the electrodes are wound together with or laminated on a separator to produce an electrode element. The electrode element is inserted into a battery case such as a can, and an electrolyte is injected into the case in fabricating a rechargeable lithium battery. The separator may be a resin such as polyethylene or polypropylene.

[0032] The following Examples further illustrate the present invention in detail, but are not to be construed to limit the scope thereof.

EXAMPLE 1

[0033] 94 wt % of a $LiCoO_2$ positive active material, 3 wt % of a Super-P conductive material, and 3 wt % of a polyvinylidene fluoride binder were mixed in a N-methylpyrrolidone solvent to prepare a positive active material slurry. The positive active material slurry was coated on a Al-foil current collector and dried to produce a positive electrode with a width of 4.9 cm and a thickness of 147 μm .

[0034] 99.9 wt % of an artificial graphite material (FHS, available from Japan Carbon Ltd.), 0.2 wt % of an oxalic acid additive, and 10 wt % of polyvinylidene fluoride binder were mixed in a N-methylpyrrolidone solvent to prepare a negative active material slurry. The negative active material slurry was coated on a Cu-foil current collector and dried to produce a negative electrode with a width of 5.1 cm and a thickness of 178 μm .